PREPARATION, CHARACTERIZATION AND PERFORMANCE EVALUATION OF LANTHANUM ORTHOFERRITES FOR HUMIC ACID REMOVAL VIA PHOTOCATALYSIS

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DEDICATION

Dedicated to my parents,
(Yahya Lim and Zubaidah Johari)
my beloved siblings,
(Norhayati, Noraini, Norhidayah, Norizzati, Syafiq and Ikmal)
family and friends who gave me inspiration, encouragement and endless support
throughout the success of my study.
May this thesis be an inspiration and guidance in the future.

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ABSTRACT

Humic acid (HA) is categorised as a natural organic matter (NOM). Excessive concentrations of HA present in water treatment system may lead to adverse effects such as undesirable taste, effluent coloration towards and production of carcinogenic by-products such as trihalomethanes. One promising way of HA elimination is by photocatalysis. Lanthanum orthoferrites ($LaFeO_3$) has been regarded as an efficient visible-light driven photocatalyst due to its narrow band gap. In order to synthesize $LaFeO_3$ nanoparticles, chelation is one of the important chemical processes to form the interaction between metals that directly affects the physicochemical properties of the nanoparticles. One of the common chelating agents used is glucose. However, synthesized $LaFeO_3$ face issues in their physicochemical properties such as low surface area and poor morphology when glucose is used as the main chelating agent. Therefore, the effect of citric acid addition on glucose as a secondary chelating agent was investigated in this study. Interestingly, after the addition of citric acid (LFO2), the BET surface area dramatically increased from 15.68 m^2/g to 40.77 m^2/g. The field emission scanning electron microscopic (FESEM) images showed that LFO2 possesses a better spherical-shaped like growth and less agglomeration. More importantly, results revealed that LFO2 degraded 80% of HA within 120 minutes, which is a 1.3-fold increment compared to LFO1 (glucose only). Furthermore, the effects of different calcination temperatures (400 °C, 500 °C and 600 °C) were also investigated using glucose and citric acid as a dual chelating agent. From the study, $LaFeO_3$ nanoparticles calcined at 400 °C were selected as the most promising photocatalyst due to its amorphous nature which benefits from the presence of surface defect. In addition, the amorphous $LaFeO_3$ also recorded the highest surface area with a value of 70.02 m^2/g which contributed to the enhancement of photocatalytic activity for the degradation of HA. Besides that, effect of operational parameters such as photocatalyst loading (0.6–1.20 g/L), initial concentration of HA (10-40 mg/L) and aeration (presence of oxygen) for HA degradation under visible light irradiation were studied using the amorphous $LaFeO_3$. Overall, the optimal values for degradation of HA were observed to be at a catalyst loading of 1.0 g/L and initial concentration of 10 mg/L. The result also showed that the presence of oxygen as electron acceptor from aerated samples preventing recombination of electrons and holes, thus enhancing the photocatalytic degradation. In a nutshell, the perovskite based-photocatalyst, $LaFeO_3$ was successfully synthesized using glucose and citric acid as a dual chelating agent assisted by low temperature calcination.

ABSTRAK

Asid humik (HA) dikategorikan sebagai bahan organik semulajadi (NOM). Kepekatan berlebihan HA di dalam sistem rawatan air boleh membawa kepada kesan buruk seperti rasa yang tidak diingini, warna efluen dan penghasilan produk sampingan karsinogenik seperti trihalometana. Salah satu cara yang boleh menjanjikan prosess penghapusan HA adalah dengan fotopemangkinan. Lantanum ortoferit ($LaFeO_3$) telah dianggap sebagai fotomangkin yang mempunyai keberkesanan di bawah cahaya nampak disebabkan oleh sela tenaga yang rendah. Untuk mensintesis nanopartikel $LaFeO_3$, pengkelatan adalah salah satu proses kimia yang penting untuk membentuk interaksi antara logam yang secara langsung mempengaruhi sifat fizikokimia nanopartikel. Salah satu agen pengkelat yang biasa digunakan ialah glukosa. Walau bagaimanapun, $LaFeO_3$ yang telah disintesis berdepan dengan beberapa isu pada sifat-sifat fizikokimianya seperti luas permukaan yang rendah dan morfologi yang lemah apabila glukosa digunakan sebagai agen pengkelat utama. Oleh itu, kesan penambahan asid sitrik pada glukosa sebagai agen pengkelat kedua telah disiasat dalam kajian ini. Menariknya, selepas penambahan asid sitrik (LFO2), luas permukaan BET didapati telah meningkat secara mendadak daripada 15.68 m^2/g ke 40.77 m^2/g. Mikroskop elektron imbasan pancaran medan (FESEM) menunjukkan bahawa LFO2 mempunyai morfologi yang lebih baik dari segi pembentukan sfera yang lebih sekata dan kurang penggumpalan. Lebih penting lagi, keputusan mendedahkan bahawa LFO2 mampu menguraikan 80% HA dalam tempoh 120 minit, iaitu kenaikan 1.3 kali ganda berbanding LFO1 (glukosa sahaja). Selain itu, kesan suhu pengkalsinan yang berbeza (400 °C, 500 °C dan 600 °C) juga telah dikaji menggunakan glukosa dan asid sitrik sebagai agen pengkelat dwi. Daripada kajian ini, suhu pengkalsinan nanopartikel $LaFeO_3$ pada 400 °C telah dipilih sebagai fotomangkin yang paling berpotensi disebabkan oleh sifat amorfusnya iaitu mendapat faedah daripada kecacatan pada permukaannya. Tambahan pula, $LaFeO_3$ amorfus juga mencatatkan luas permukaan yang paling tinggi dengan nilai 70.02 m^2/g yang menyumbang kepada peningkatan aktiviti fotopemangkinan untuk penguraian HA. Di samping itu, kesan parameter operasi seperti muatan pemangkin yang digunakan (0.6-1.20 g/L), kepekatan awal HA (10-40 mg/L) dan kesan pengudaraan (kehadiran oksigen) untuk penguraian HA di bawah radiasi cahaya nampak telah dikaji menggunakan amorfus $LaFeO_3$. Secara keseluruhan, nilai optimum bagi penguraiaan HA diperhatikan adalah pada muatan mangkin sebanyak 1.0 g/L dengan kepekatan awal HA sebanyak 10 mg/L. Keputusan juga menunjukkan kehadiran oksigen sebagai penerima elektron daripada sampel yang diudarakan menghalang daripada penggabungan semula lubang-elektron, lalu meningkatkan prosess penguraian fotobermangkin. Secara ringkasnya, fotomangkin berasaskan bahan perovskit, $LaFeO_3$ telah berjaya disintesis dengan menggunakan glukosa dan asid sitrik sebagai agen pengkelat dwi dan dibantu oleh suhu pengkalsinan yang rendah.

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LIST OF ABBREVIATIONS

AOPs	-	Advanced oxidation processes
BET	-	Brunauer–Emmett–Teller
BJH	-	Barrett–Joyner–Halenda
CA	-	Citric acid
DI	-	Deionized water
EDX	-	Energy-dispersive X-ray
EDTA	-	Ethylenediaminetetraacetic acid
FESEM	-	Fourier electron-scanning electron microscope
FTIR	-	Fourier transform infrared
HA	-	Humic acid
IUPAC	-	International union of pure and applied chemistry
$LaFeO_3$	-	Lanthanum orthoferrites
N_2	-	Nitrogen gas
SEM	-	Scanning electron microscope
TiO_2	-	Titanium Dioxide
TEM	-	Transmission electron microscopy
TOC	-	Total organic carbon
UV	-	Ultraviolet
XRD	-	X-ray powder diffraction

LIST OF SYMBOLS

Mg	-	Milligram
L	-	Litre
°C	-	Degree celcius
µm	-	Micrometre
M	-	Meter
nm	-	Nanometer
g	-	Gram
%	-	Percentage
M	-	Molarity
Ppm	-	Part per million
$[HA]_o$	-	Initial concentration of humic acid
w/v	-	Weight per volume
Min	-	Minutes
K	-	Rate constant
R^2	-	Linear regression
S	-	Second
W	-	Watt
T	-	Times
A	-	Lambda

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CHAPTER 1

INTRODUCTION

1.1 Research Background

Humic acid (HA) is a major component of natural organic matter (NOM). The formation of HA is usually initiated by the breakdown of animal carcasses and plant residues and can be vastly found in the aquatic system, including surface and ground water (Fabris *et al.*, 2008). In general, HA is composed of hydrophilic portions, consisting of OH^- groups, and hydrophobic portions, consisting of aliphatic chains and aromatic rings. However, the structure is more prevalent with phenolic and carboxylic groups. In the past decades, the penetration of HA into water sources via drainages into water bodies are worryingly increasing. According to experts, climate change is the main factor that contributes to this increment (Bhatnagar and Sillanpää, 2017; Wang *et al.*, 2002). It is known that floods, droughts, rainfalls, snowmelt runoffs are example of events which are resulted from climate changes (Nkambule *et al.*, 2012). Moreover, it has been reported that visible impact on the quality of water is to be expected by the combination of these events (Hirabayashi *et al.*, 2008). High concentration of HA commonly leads to problems such as undesirable taste, coloration of drinking water, membrane fouling and formation of biofilms in pipe lines (Korotta-Gamage and Sathasivan, 2017; Lu *et al.*, 2016). However, the utmost impact of HA is when it interacts with disinfectants used in drinking water treatment process. (Mohora *et al.*, 2012). The reaction of HA with chlorine during disinfection would produce carcinogenic by-products like trihalomethanes which turns out to be a major problem for drinking water treatment (Kim *et al.*, 2017; Serrano *et al.*, 2015).

Despite many conventional treatment techniques such as adsorption (Bhatnagar and Sillanpää, 2017), coagulation (Matilainen *et al.*, 2010), and biological treatment (Yang *et al.*, 2018) suggested for removal of HA, these contaminants are not properly removed. By considering the disadvantages of these methods, heterogeneous photocatalysis is an effective alternative solution for elimination of HA from aqueous solution. Heterogeneous photocatalysis is a catalytic process in which the reactant and the catalyst are in different phase, where the photocatalyst is typically presents as solid with the reaction taking place at the interface between phases, i.e., solid-liquid or solid-gas. The concept of photocatalysis is originally adapted from advanced oxidation processes (AOPs) where the idea is basically releasing hydroxyl radicals, •OH (Comninellis *et al.*, 2008). The hydroxyl radical is generates after the catalyst being excited by any irradiation sources of light (De Lasa and Ibrahim, 2004; Prier *et al.*, 2013). The generated hydroxyl radicals then turn into highly active species which are responsible for destruction of humic acid and further mineralize into CO_2 and H_2O.

Since the discovery of TiO_2 as a viable photocatalyst in 1972 by Fujishima and Honda, much fundamental progress has been made in developing novel semi-conductor photocatalysts such as WO_3 (Maghsoodloo *et al.*, 2011), ZnO (Zhao *et al.*, 2011), CuS (Vakili *et al.*, 2014), BiOBr (Sillanpää *et al.*, 2018), $BiFeO_3$ (Wei *et al.*, 2018) and others, particularly visible light response catalysts for efficient utilization of solar energy. Recently, perovskite based-photocatalyst is discovered to be an efficient photocatalyst due to its unique crystal structures and electronic properties under visible light irradiation. There are different types of perovskite that has been further explored, such as titanite perovskite (Itoh *et al.*, 1999), tantalite perovskite (Machida *et al.*, 2000), vanadium-based perovskite (Dang and Millis, 2013) and ferrite perovskite (Liu *et al.*, 2010). Lanthanum orthoferrites ($LaFeO_3$) is regarded as a an efficient visible-light driven photocatalyst for photocatalytic reaction due to its narrow band gap and optoelectronic properties (Li *et al.*, 2015; Thirumalairajan *et al.*, 2012).

In order to produce $LaFeO_3$ of desired surface area and morphology, there are few important criteria's that are to be considered, such as synthesis methods, chelating agents, calcination temperature and others (Cecchini *et al.*, 2014; Chen *et al.*, 2008). Based on literature, sol-gel, hydrothermal, co-precipitation and gel-combustion methods are frequent method for synthesizing $LaFeO_3$ due to the ability to produce photocatalyst of desired properties (Augustin and Kalaiselvan, 2003; Das and Kandimalla, 2017). Gel-combustion is regarded as the most adaptable method due to its simplicity, cost-efficiency and large scale produce compared to hydrothermal and co-precipitation method for the synthesis of $LaFeO_3$ (Deganello and Tyagi, 2018; Hao and Zhang, 2017). In order to synthesize $LaFeO_3$ nanoparticles in gel-combustion method, chelation is one of the chemical processes that gives influence to the strength of metal–ligand interactions. In chelate formation, chelating agents are typically act as intermediate substance that interact intimately with metal cations and thus, providing stable gel network formation during the synthesizing process (Kołodyńska, 2011; Priyadharsini *et al.*, 2018; Samoila *et al.*, 2012). In this sense, many chemical compounds have been proposed as chelating agents.

On the other hand, calcination temperature also is regarded as a key parameter in the enhancement of photocatalytic activity (Ali *et al.*, 2018; Shen *et al.*, 2016). This is because thermal treatment has a prominent influence on the physicochemical properties of photocatalyst (Pereyma *et al.*, 2018). It is known that calcination temperatures could affect the physicochemical properties such as band gap, crystallinity aside from surface area and morphology properties (Cen *et al.*, 2014; Shen *et al.*, 2016). All these properties will determine the efficiency of the photocatalyst.

1.2 Problem Statements

In gel combustion method, chelating agent plays significant roles during the synthesizing process which results in the uniform sol solution, and facilitates the formation of homogeneous nanoparticles. Theoretically, chelating agents act as a binder to form interaction between metals that directly affects the physicochemical properties of the nanoparticle. Without the presence of chelating agents, the nanoparticles will be resulted as severe agglomeration in morphology, low surface area and thus, affected the photocatalytic performance (Siwińska-Stefańska *et al.*, 2015). Recently, many of studies attempt to investigate the effect of different of chelating agents on the physicochemical properties of nanoparticles (Kołodyńska, 2011; Samoila *et al.*, 2012; Tabesh *et al.*, 2017). It is known that different chelating agents have its own functional group that subsequently exhibit different interactions in chelation process. One of the common chelating agent used in previous studies is glucose (Ansari *et al.*, 2018; Liu and Xu, 2011). Glucose is known as eco-friendly source, low cost, facile and reproducibility. Most importantly, using glucose as the chelating agents often leads to convenience method which requires less energy for synthesis of high purity nanoparticles. However, glucose act as a single chelating agent is not able to accommodate all the metals completely due to the weak electron donating group and subsequently produces $LaFeO_3$ with low surface area and high agglomeration degree (Li *et al.*, 2015; Priyadharsini *et al.*, 2018). Therefore, presence of secondary chelating agent is essential in order to improve the interaction between the metal cation so that the stability of metal oxide can be achieved. However, the effect of secondary chelating agent on the physicochemical properties of $LaFeO_3$ by the gel-combustion method has been scarcely investigated. Thus, this work aims to employ citric acid (CA) as a secondary chelating agent that can possibly improve the physicochemical properties of $LaFeO_3$ due to its highly structural stability and ability to build good interaction with the primary chelating agent which somehow can lead to better photocatalytic activity.

Besides that, calcination temperature is another vital parameter that will affect the surface area and morphological properties of $LaFeO_3$. According to literature, increase of calcination temperature will also affect physicochemical properties such as band gap, crystallinity aside from surface area and morphology (Ali *et al.*, 2018; Shen *et al.*, 2016). One way or another, many researchers have outlined the fact that high calcination temperatures often produce photocatalyst of high crystallinity and better morphology compared to low calcination temperatures (Hakki *et al.*, 2018; Klaysri *et al.*, 2015). However, it is known that increasing the thermal treatment temperature would consume higher energy and leads to higher operation cost (Gasia *et al.*, 2017; Smith *et al.*, 2018). In this case, it would not be efficient to be adapted in industry. Lately, approaches using amorphous structures have started to gain attention due to its lower synthesis cost and facile scaling up (Korotcenkov, 2008; Yao and He, 2014). Moreover, amorphous material has concisely exhibit large surface area, something important for an efficient photocatalyst (Castillo *et al.*, 2010; Wang *et al.*, 2017; Yoon and Cocke, 1986). In addition to that, amorphous materials also provide active centres for charge separation via the production of surface defects (impurities, micro voids, and oxygen vacancies) and subsequently, promotes high absorption of visible light as compared to crystalline materials (Ma *et al.*, 2014). Apart from that, the operational parameter for photocatalytic degradation such as catalyst loading, initial concentration of HA and oxidizing agents is further investigated to establish highly efficient photocatalytic degradation of HA.

1.3 Research Objectives

The aim of this study is to develop a perovskite-based photocatalyst, $LaFeO_3$ for the photocatalytic degradation of humic acid present in water treatment. The specific objectives for this study are as follows:

1. To identify the effects of citric acid addition as a secondary chelating agent on the physicochemical properties of the synthesized $LaFeO_3$ photocatalyst.
2. To determine the influences of calcination temperatures (400 °C, 500 °C and 600 °C) on the physicochemical properties of the synthesized $LaFeO_3$ photocatalyst.
3. To evaluate the effects of operating parameters such as catalyst loading, initial concentration and presence of oxygen as an oxidizing agent on the photocatalytic degradation efficiency of humic acid.

1.4 Research Scopes

In order to achieve the objectives of this study, the following tasks of work were conducted:

1. Synthesizing $LaFeO_3$ via gel combustion method by varying two routes namely as glucose method and glucose-citric acid method.

2. Characterization of the physicochemical properties of the synthesized $LaFeO_3$ in terms of thermal stability, morphological properties, surface areas, crystallinity and optical properties using Thermogravimetric analysis (TGA), Field Emission Scanning Electron Microscope (FESEM), Energy-dispersive X-ray spectroscopy (EDX), Brunauer–Emmett–Teller (BET) surface area, X-ray powder diffraction (XRD) and UV-Vis spectrophotometer.

3. Evaluation of the photocatalytic degradation performance of HA under visible light irradiation (100 Watt LED) using synthesized $LaFeO_3$ in order to prove adding citric acid as secondary chelating agent on glucose can have better performance.

4. Synthesis of $LaFeO_3$ using dual chelating agents, glucose and citric acid by varying the calcination temperature at 400 °C, 500 °C and 600 °C in order to study the influences of calcination temperatures on the physicochemical properties of $LaFeO_3$ photocatalyst.

5. Characterization of the synthesized photocatalyst using FESEM, EDX, BET surface area, XRD and UV-Vis spectrophotometer.

6. Evaluation of the photocatalytic activity i.e degradation of HA under visible light irradiation (100 Watt LED) using synthesized $LaFeO_3$ from different of calcination temperature (400 °C, 500 °C and 600 °C).

7. Evaluation of photocatalytic activity for the degradation of HA under visible light irradiation (100 Watt LED) using selected photocatalyst (400 °C) by varying the operating parameters conditions. The operating parameters are catalyst loading, initial concentration of HA and the effect of oxygen via aeration (air flow rate of 0.62 L/min.kg) as an oxidizing agent.

1.5 Significance of Study

Photocatalysis, is an effective method to oxidize many organic contaminants at ambient conditions. Thus, researches on synthesizing visible-light driven photocatalyst that are easy to be produced and large scalable nanoparticles with desired properties are the main priorities. In this study, rapid production of high surface area $LaFeO_3$ photocatalyst via gel-combustion method using the addition of citric acid to glucose as a secondary chelating agent were presented. Citric acid was considered as a secondary chelating agent in this study due to its high structural stability and builds good interaction with the primary chelating agent. Additionally, the effect of calcination temperature on physiochemical properties of synthesized photocatalyst was also studied upon as researches seek ways to produce photocatalyst at lower cost and energy consumption. In fact, this study proves that higher calcination temperatures impart poor physicochemical properties onto photocatalyst which resulted low performance of photocatalytic activity. The synthesized $LaFeO_3$ photocatalyst via this new approach together with ideal calcination temperature and optimum photocatalytic operating conditions led to significant improvement in photocatalytic activity for the degradation of humic acid under visible light irradiation.

1.6 Organization of the Thesis

This thesis consists of 5 chapters. Chapter 1 provides a brief information on humic acid contaminants and the current issues plaguing humic acid removal. The objectives, scopes and the research significance were also highlighted in this chapter. In Chapter 2, literature review done for this research work, including detailed information of humic acid such as its chemical structure and the adverse effects of excessive humic acid in water treatment plants. Furthermore, the chapter also discussed in detail on the important criteria's such as synthesis method, chelating agents and calcination temperature that were considered in order to synthesize photocatalyst with highly desired properties. The background and reason for selecting $LaFeO_3$ was also highlighted. The proposed mechanism of chelating based on previous studies were explained in detail. Lastly, the chapter discussed the fundamental basics of operational parameter for photocatalytic degradation of humic acid. In Chapter 3, a reliable pathway for preparation of $LaFeO_3$ via gel combustion method was described. Various characterization tools that were employed to define the characteristics of synthesised photocatalyst were also presented. Finally, the photocatalytic activity of synthesized were discussed.

Results and discussion were deliberated in Chapter 4. In this chapter, detail effects of citric acid on glucose as a secondary chelating agent on the physicochemical properties of synthesized $LaFeO_3$ photocatalyst has been discussed in detail. The improvement in physicochemical properties of $LaFeO_3$ using the proposed mechanism were described based on the chelation process between the functional group and metal cations. Also in this chapter 4, the influences of calcination temperatures (400 °C, 500 °C and 600 °C) on the physicochemical properties of synthesized $LaFeO_3$ photocatalyst has been further deliberated. Optimum calcination temperature was selected and was further discussed in more detail. The effects of operational parameters such as catalyst loading, initial concentration and presence of oxygen as an oxidizing agent on the photocatalytic degradation efficiency of HA were detailed. Based on the photocatalytic degradation performance, the optimal value from each parameter were selected. Finally, a general conclusion of this study and some recommendation for future work were listed in Chapter 5.

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